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In re International Application of

Kazuo FUJIURA, Tadayuki IMAI, Masahiro SASAURA and
Kouichirou NAKAMURA

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September 26, 2006

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Atsuhiko HAMANAKA

APPLICATION FOR UNITED STATES LETTERS PATENT

INVENTOR(S): Kazuo FUJIURA
 Tadayuki IMAI
 Masahiro SASAURA
 Kouichirou NAKAMURA

INVENTION: AN OPTICAL MEDIUM, AN OPTICAL LENS AND A PRISM

S P E C I F I C A T I O N

DESCRIPTION

AN OPTICAL MEDIUM, AN OPTICAL LENS AND A PRISM

5 Technical Field

[0001] The present invention relates to an optical medium, an optical lens and a prism, and more particularly relates to an optical medium, an optical lens and a prism having a high refractive index without anisotropy and a wide passband.

10

Background Art

[0002] Conventionally, optical components such as an optical lens and a prism are used for optical apparatuses such as a camera, a microscope and a telescope; electronograph
15 system recording apparatuses such as a printer and a copier; an optical recording medium such as DVD; and an optical device. For example, it is necessary to make the beam diameter of a laser beam for recording as small as possible so as to raise recording density in the field of an optical recording. Thus,
20 in order to focus shorter wavelength light efficiently, a lens and a prism that retain high optical transmittance to a short wavelength band with a high refractive index, without anisotropy, is necessary.

[0003] For example, the beam spot diameter of a laser
25 beam is determined by a wavelength λ of a light source and a numeric aperture NA of a lens, and is known as $0.8 \times \lambda/NA$. An information capacity of 4.7GB can be recorded in a

conventional DVD recording apparatus having a 5-inch disk using a semiconductor laser with a 650nm wavelength and a lens having $NA=0.6$. Recently, DVD (Blu-ray) recording apparatuses with enlarged NA and decreased wavelength have been developed. These apparatuses have realized a storage capacity of about 23GB in a 5-inch disk using a semiconductor laser with a 405nm wavelength and a lens having $NA=0.85$ as a light source.

[0004] Additionally, a near field recording method is known to raise recording density by Evanescent light at the reflecting surface using a minute lens with a high refractive index. This minute lens is a hemispheric lens called SIL (Solid Immersion Lens) and is placed between an optical recording medium and an objective lens. In such an optical system, the beam spot diameter of a laser beam transmitted through the objective lens equivalently becomes $\lambda/(nk \times NA)$ (n is the refractive index of a SIL) and can focus to $1/n$ compared to those without a SIL. (e.g., See Nonpatent Document 1). In the area where the gap between the recording surface of the optical recording medium and the bottom surface of the SIL is below $1/4$ of the light wavelength, a laser beam transmitted through the SIL is outputted in a same disposition as an interior of the SIL. Then, the beam spot diameter is focus to $1/n$ of a diffraction limit.

[0005] Above referenced SIL becomes $NA=n^2\sin\theta$ when incident angle is θ and has a great influence on the refractive index of an optical medium. For this reason, the optical

medium with a high refractive index is crucial and an isotropic optical medium without birefringence is crucial at focal power. In addition, it is important to be an optical medium whose optical transmittance does not deteriorate to the short wavelength band. Beside a SIL, optical components such as a camera, a microscope and a stepper have implemental constraint and, a small lens with a high refractive index and high focal power is necessary. If the lens is of the same size, large NA, high focal power and bright lens is preferable. In the same way, a prism may be realized small and sufficient spectroscopic characterization by using high refractive index materials.

[0006] Concerning the above mentioned viewpoint, high refractive index glasses and crystal materials have been examined. For the glasses, high refractive index glasses containing much La and Pb, and TeO₂-based glasses are known. However, an optical medium that can realize a refractive index of 2.2 in a visible wavelength band has not been developed. There is a problem in that optical transmittance at around 400nm deteriorates by raising refractive index.

[0007] Meanwhile, concerning the crystal materials, a lot of lenses with a high refractive index have been developed by using oxide crystals (e.g., See Patent Document 1). However, optically isotropic materials without birefringence are limited to cubic crystal structure. Although, many crystals are disclosed in the Patent Document 1, isotropic materials are limited to SrNbO₃, SrTaO₃, Bi₂₀SiO₁₂, Bi₂₀GeO₁₂, Bi₄Ge₃O₁₂

and GaP. Concerning these crystals, the refractive index in the visible wavelength band is from 2.06 to 2.22.

[0008] In addition, high-intensity liquid crystal projectors require a prism for a polarization optical system, which has high optical transmittance of the visible wavelength band without anisotropy of refractive index, and has a high refractive index. Although borosilicate glasses were used conventionally, there was a disadvantage that photo-elastic effect is large. Thus, it is examined to use high refractive index glasses such as lead-containing glasses. However, there has been a problem that these glasses have low transmittance in the short wavelength band, and cannot be used in the wavelength band used for the liquid crystal projector.

[0009] Furthermore, an air pollution measuring device has no absorption up to a long wavelength band of around $5\mu\text{m}$, and requires optical materials with a high refractive index. Conventionally, optical transmittance in the long wavelength band of the optical medium is as follows. An optical transmission range of silica based glasses is up to around $2\mu\text{m}$ and silica based glasses have a low refractive index. Fluoride glasses such as ZBLAN ($\text{ZrF}_4\text{-BaF}_2\text{-LaF}_3\text{-AlF}_3\text{-NaF}$) have high optical transmittance but the refractive index is low around 1.5. Chalcogenite glasses such as Ge-Sb-Se have high optical transmittance but have problem in toxicity. Therefore, optical medium with high optical transmittance in the long wavelength band and a high refractive index is

required.

[0010] Patent Document 1: Japanese Patent Application
Laying-Open No. 2000-19301

Nonpatent Document 1: Sharp Technical Report "Trend of
5 high-capacity optical disk" Vol. 72, December, 1998, pp.9-12

Disclosure of the Invention

[0011] An object of the present invention is to provide
an optical medium, an optical lens and a prism having a high
10 refractive index without anisotropy and a wide passband.

[0012] In order to accomplish such the object, an optical
medium of the present invention consists of a cubic crystal
material. The crystal material is $\alpha\beta\text{O}_3$, where α is at least
one of K, Ba, Sr, Ca, and β is at least one of Ta, Ti. For
15 example, when α is K, and β is Ta, a high refractive index
of 2.2-2.4 in a visible wavelength band without birefringence
in a wide temperature range can be attained. Additionally,
the crystal material is KTaO_{3-d} , where the amount of oxygen
deficiency d is $0 \leq d < 10^{-7}$.

20 [0013] In other aspect, the crystal material is $\text{KTa}_{1-x}\text{Nb}_x\text{O}_3$,
where composition x is $0 \leq x \leq 0.35$. This composition enables
to raise refractive index while its phase transition
temperature is below a room temperature. In addition, the
crystal material is $\text{K}_{1-y}\text{Li}_y\text{TaO}_3$, where composition y is
25 $0 \leq y \leq 0.02$.

[0014] In the other aspect, the crystal material is
 $\text{K}_{1-y}\text{Li}_y\text{Ta}_{1-x}\text{Nb}_x\text{O}_3$, where composition y is $0 \leq y \leq 0.02$. This

composition enables a crystal phase transition to be the second order phase transition without latent heat and may settle problems such as a crack formation.

5 Brief Description of the Drawings

[0015] FIG. 1 is a view illustrating wavelength dependence of KT refractive index;

 FIG. 2 is a view illustrating optical transmittance of KT;

10 FIG. 3 is a view illustrating the relationship between oxygen deficiency of KT and absorption coefficient at 405nm;

 FIG. 4 is a view illustrating a pickup system of DVD recording apparatus;

 FIG. 5 is a view illustrating the relationship between
15 Nb content x of KTN, refractive indices, and phase transition temperatures;

 FIG. 6 is a view illustrating the relationship between Nb content x of KTN, short wavelength absorption edges, and Abbe numbers;

20 FIG. 7 is a view illustrating a spectrum of KTN at the long wavelength band;

 FIG. 8 is a view illustrating the relationship between Sr content x and phase transition temperatures;

 FIG. 9 is a view illustrating the composition of a
25 cross-dichroic prism according to one embodiment of the present invention;

Best Modes for Carrying Out the Invention

[0016] Embodiments of the present invention will be described in detail with reference to the drawings. The present embodiments are characterized by producing lenses
5 and prisms having composition to be cubic crystal at a room temperature by employing crystal materials of chemical formula $K_{1-y}Li_yTa_{1-x}Nb_xO_3$ ($0 \leq x \leq 1, 0 \leq y \leq 1$, hereinafter called KLTN) as optical materials. KLTN has the nature to change a crystal system from a cubic to a tetragonal depend on
10 temperatures. When Li content is in the range of 0-0.02, and Nb content is in the range of 0-0.35, a phase transition temperature of a cubic to a tetragonal may be set below a room temperature. Therefore, the optical medium without birefringence may be obtained in the usage at a room temperature.
15 Then, the lens and the prism produced by these crystal materials have negligible small polarization dependency of transmitted light.

[0017] (KT)

In the above composition, $KTaO_3$ ($x=0, y=0$, hereinafter
20 called KT) has a phase transition temperature of approximately -273°C and crystal materials without birefringence may be obtained in a wide temperature range. In this composition, high refractive indices from 2.2 to 2.4 may be obtained in the visible wavelength band and KT has high performance as
25 optical lenses and prisms. FIG.1 shows wavelength dependence of KT refractive index, and FIG.2 shows optical transmittance of KT. $KTaO_3$ has refractive indices of 2.2 or more in the

visible wavelength range (400–800nm), and reaches 2.38 at around 400nm. In addition, the optical transmittance shown in FIG. 2 has a light absorption edge of approximately 360nm. It is clear that sufficient light transparency to the short wavelength is maintained. In particular, an optical material with 10mm-thick has transmittance of 80% or more at around 400nm.

[0018] However, a perovskite oxide has oxygen deficiency easily depending on manufacturing condition and heat

treatment condition. The light absorption due to carriers generated by this oxygen deficiency deteriorates optical transmittance. FIG.3 shows the relationship between oxygen deficiency of KT and an absorption coefficient at 405nm. When developing KTaO_3 crystals, a sample manufactured by changing the oxygen partial pressure in atmosphere is prepared. The amount of oxygen deficiency is measured by increasing weight using thermogravimetric analysis in oxygen atmosphere and an absorption coefficient in the visible wavelength band is measured for every sample. FIG.3 shows that an absorption coefficient increases with the increase of oxygen deficiency. Considering practicality of a lens and the like, internal optical transmittance of more than 90% per cm is desirable. Thus, it is indispensable that oxygen deficiency of KT below 10^{-7} where KT composition is represented as KTaO_{3-d} ($0 \leq d < 10^{-7}$) in that case.

[0019] In addition, K included in crystal materials may be replaced with an element at least one of Ba, Sr and Ca,

and Ta may be replaced with Ti. Even these crystals may raise refractive index without substantial changes in phase transition temperatures.

[0020] (KTN)

5 Furthermore, in the cases when a high refractive index material is necessary, refractive indices can be raised effectively by adding Nb ($\text{KTa}_{1-x}\text{Nb}_x\text{O}_3$; $0 \leq x \leq 1$, $y=0$, hereinafter called KTN). However, a phase transition temperature becomes approximately 420°C at KNbO_3 ($x=0$, $y=0$) and KTN has
10 birefringence at a room temperature. Therefore, at a room temperature, Nb content has limitation. In particular, the phase transition temperature rises above the room temperature when Nb content exceeds 35% against Ta. Moreover, when a storage temperature and a transport temperature are below
15 the phase transition temperature, crystal materials repeat phase transition even the phase transition temperature is below the room temperature. In this instance, structural change of crystal is accompanied and a factor of reliability degradation such as cracks in crystals occurs. This is because
20 a phase transition is the first order phase transition with latent heat, in KTN composition.

[0021] (KLTN)

Consequently, by adding Li, a phase transition is the second order phase transition with no latent heat. This may
25 solve the problem of cracks. Therefore, when a phase transition temperature is low enough compared to a storage temperature and a transport temperature, lenses and prisms

composed by KTN have sufficiently high-efficiency and high reliability. In addition, KLTN being added to Li becomes effective, when refractive indices increase and both storage and transport temperatures have approached to a phase transition temperature.

[0022] Based on the above, optical lenses and prisms with a high refractive index without birefringence may be realized. The following examples illustrate some preferred modes of the present invention, but are not intended to limit the scope of the claimed invention.

Example 1

[0023] (lens consists of KT crystals)

KT crystals developed by TSSG method is sliced to 1.2-1.5mm thickness in [100] direction with a wire saw. The sliced substrate is cut at intervals of 1.2-1.5mm using the wire saw to produce a cube of 1.2-1.5mm. By putting this cubic crystal into a container with polishing materials and agitate to round off the corners to produce an almost spherical rough grinded ball. A ball lens having a diameter of 1.0 mm is obtained by putting this rough grinded ball, polishing materials between two grinding substrates and spinning with a constant weight. A hemispheric lens is obtained by fixing a ball lens on grinding substrates with a wax, and by rotating to grind with a constant weight to smooth one surface. A pickup of a DVD recorder is composed by using this minute hemispheric lens as a SIL.

[0024] FIG.4 shows a composition of the pick-up system of a DVD recorder. The laser beam emitted from a semiconductor laser is focused to a predetermined beam spot diameter through objective lens 31. The emitted laser beam from objective lens 5 31 is collected at SIL32 and focused at the bottom of SIL32. An interval between the recording surface of optical recording medium 33 and the bottom of SIL32 is set to $1/4$ or less the light wavelength. The laser beam leaked from SIL32 reaches optical recording medium 33 at the predetermined beam spot 10 diameter.

[0025] The semiconductor laser referenced in Example 1 has a wavelength of 685nm. The DVD recorder has an objective lens of NA=0.65 and a SIL of KT crystal at a refractive index of 2.23. The recording density of the DVD is measured by an 15 evaluation equipment for DVD recording, the recording density of 19Gbit/inch² may be realized. When a conventional high refractive index glass is used for a SIL, a refractive index at 685nm is 2.0 and a recording density stays at 16Gbit/inch².

[0026] Therefore, using a lens of which refractive index 20 is higher than that of conventional lenses, high recording density may be realized. Also, according to lens materials described in Example 1, it is clear that the materials have superior in focal power and has no birefringence of materials.

[0027] Although the present example describes the 25 semiconductor laser with a 685nm wavelength, lens materials of the present example are preferred to up to a 360nm wavelength in focal power. Consequently, a higher density recording is

realizable in the short wavelength band. For example, using an evaluation equipment for DVD recording having a semiconductor laser with a 405nm wavelength and a SIL of NA=2.2, a recording capacity of 150GB may be realized by applying near-field recording to a 5-inch disk.

Example 2

[0028] (lens consists of KTN crystals)

FIG.5 shows relationships between Nb content x of KTN, refractive indices and phase transition temperatures. The measurement wavelength is 632.8nm. Refractive indices rise in proportion to the amount of Nb content, and reach a refractive index of 2.27 at $x=0.35$. A phase transition temperature and a refractive index rise linearly and reach approximately 25°C at $x=0.35$. Consequently, it is clear that Nb content is useful for increasing refractive index, and important to keep Nb content below 0.35 in order to use under the condition without birefringence of crystal.

[0029] A SIL is produced as described in Example 1 by using a crystal of Nb content $x=0.35$. The DVD recorder has an objective lens of NA=0.65, and a SIL of KTN crystal at a refractive index of 2.27. The recording density of the DVD recorder is measured by the evaluation equipment for DVD recording, the recording density of 21Gbit/inch² may be realized. In addition, using an evaluation equipment for DVD recording having a semiconductor laser with a 405nm wavelength and a SIL of NA=2.2, a recording capacity of 160GB may be

realized by applying near-field recording to a 5-inch disk.
[0030] Meanwhile, when Nb content exceeds 0.35, a phase transition temperature of crystals approaches a room temperature. Then, a phase transition to tetragonal easily
5 occurs in association with external influences such as pressure to crystals, so that optically isotropic materials are not obtained. In addition, light transmission characteristics deteriorate and a wavelength absorption edge becomes 400nm. Therefore, when the amount of Nb content
10 becomes 0.35 or more, it is possible to make a lens of NA increased, but optical homogeneity and transmission characteristics are not acquired practically. FIG.6 shows the relationships between Nb content x of KTN, short wavelength absorption edges and Abbe numbers. As refractive indices of
15 crystal containing a high proportion of Nb get higher, dispersion become significant, and a light absorption edge shifts to the long wavelength side. Moreover, refractive index dispersion becomes significant as a light absorption edge shifts to the long wavelength side, that is, Abbe number
20 becomes small. KTN materials become a high refractive index and high dispersion by addition of Nb.

[0031] FIG.7 shows a long wavelength side spectrum of KTN. Since transmittance of the vertical axis includes reflection of crystal to the both sides, it is not an internal
25 transmittance. For example, an internal transmittance at $4\mu\text{m}$ is 100%. Thus, KTN crystals are free from absorption up to a $5\mu\text{m}$ wavelength and has a high refractive index. That is

to say, it may be applied to a lens and a prism in the infrared region.

Example 3

5 [0032] (KLT)

By adding Li to KT, a crystal growth temperature can be lowered and the amount of evaporation of K_2O in the crystal growth process can be reduced. If K_2O evaporate, it condenses in low temperature area of the upper crystal manufacturing
10 device. If the amount of condensates increases, it will drop in crucible. The drop of the condensates constricts steady growth of crystal since a junk-crystal including the condensates as a core floats in a solution. Therefore, by adding Li to KT, growing in size crystal, improvement in the
15 yield and cost-reduction of lens may be realized.

[0033] By adding Li_2CO_3 in the solution of KT, $K_{1-y}Li_yTaO_3$ ($0 \leq y \leq 0.02$, hereinafter called KLT) crystal is prepared. Composition y is stated 0.02 or less because it is impossible to maintain cubic crystal at $y > 0.02$. On this
20 occasion, the amount of Li_2CO_3 against K_2CO_3 is 18mol%. The refractive index of KLT against composition y at 405nm is stated as follow.

$$n(@405nm) = 2.353 - 0.19y$$

As is clear from this formula, a crystal refractive index
25 is decreased with addition of Li. However, the decrease is only about 0.0038 when the maximum addition of Li is at 0.02 and practically acceptable. On the other hand, a crystal

growth temperature may be lowered, as mentioned above, which leads to yield improvement by 20%.

Example 4

5 [0034] In regard to crystal materials described in Example 1, BaTiO_3 , which K is replaced to Ba and Ta is replaced to Ti, has a phase transition temperature of 120°C . Consequently, as crystal materials described in Example 3, $\text{Ba}_{1-x}\text{Sr}_x\text{TiO}_3$ ($0.34 \leq x \leq 1$) with which Li is replaced to Sr is
10 produced. FIG.8 shows the relationship between Sr content x and phase transition temperatures. As Sr content increases, a phase transition temperature from cubic to tetragonal decreases. In order to keep cubic crystal at a room temperature, a phase transition temperature is desired to
15 maintain below a room temperature, and composition x is desired to be over 0.34, as shown in FIG.8.

Example 5

[0035] (KLTN)

20 As shown in FIG.5, when Nb content is over 0.2, a phase transition temperature exceeds -100°C . Therefore, a storage temperature and a transport temperature may get lower than a phase transition temperature. Then, a phase transition of crystal materials is repeated and a crack forms. To avoid
25 the crack, KLTN of Li content $y=0.01$ is employed.

[0036] Crystals of Nb content $x=0.35$ are produced, and a temperature cycle of -45°C to $+60^\circ\text{C}$ is performed.

Accordingly, crystals without Li had minute crack in 2-3 samples out of 100 samples, after 1000 cycles. On the other hand, no cracks were found in crystals with Li content $y=0.01$.

5 Example 6

[0037] (prism consists of KT crystals)

FIG.9 shows the composition of a cross-dichroic prism according to one embodiment of the present invention. By use of same constituent of KT crystal as Example 1, four triangular
10 prisms 51a-51d employed in the 3 CCD-color separation/composition optical system are produced. Conventional polishing technique is performed for the production. Dielectric multilayer film coatings are performed on right-angled side of triangular prisms 51a-51d.
15 Each right-angled side of triangular prisms 51a-51d is joined. On the right-angled side 52a, 52c, which is one of the diagonal lines of the cross-section of the joined quadrangular prism, a multilayer film which reflects R signal of RGB signal and transmits G signal and B signal is attached. On the other
20 diagonal lines as right-angled side 52b, 52d, a multilayer film which reflects B signal and transmits R signal and G signal is attached. Thus, a cross-dichroic prism used for the 3 CCD-color separation/composition optical system separates into RGB signals, modulates each signal and combines
25 the modulated signals.

[0038] This cross-dichroic prism is installed in a projector. A light source project an image by using an

extra-high-pressure mercury lamp, a metal halide lamp or a high-power xenon lamp with a high-intensity of 2000 lumens. The cross-dichroic prism described in the present example has an irradiation intensity of $2.2\text{W}/\text{cm}^2$, and has a
5 transmission deterioration of 1% or less under a 10-minute irradiation. Therefore, the projected image of the projector has no luminance transition over an extended period of time and is able to maintain image of high color rendering properties. This way, KT crystal has high optical tolerance, high
10 homogeneity, and high light transmission characteristics, hence the cross-dichroic prism which employs these materials may be applied to an imaging device of high light input such as projectors.

15 Example 7

[0039] (prism comprising KTN crystal)

The cross-dichroic prism as described in Example 6 is produced by using the KTN crystals described in Example 2. It is applied to the projector which constitutes the 3CCD-color
20 separation/composition optical system, as described in Example 6. In a prism with Nb content, optical transmission characteristics and optical tolerance do not deteriorate. The projector can project images of the stable high luminosity as Example 6. Even when KTLN crystals with Li content are
25 used, the cross-dichroic prism can maintain sufficient characteristics.

Example 8

[0040] (Polycrystalline material)

Although the crystal materials mentioned in the above Examples use single crystal materials, polycrystalline materials may also produce optical medium having a high refractive index without anisotropy and a wide transmission wavelength range. The producing method of polycrystalline KT is described in the following. After mixing powders of K_2CO_3 and Ta_2O_5 as raw materials with a ratio of 1:1, they will be heated for 10 hours in an oxygen environment at $1,000^\circ C$ inside a platinum container. $KTaO_3$ is formed with an elimination reaction of CO_2 by heating. After grounding lightly the formed $KTaO_3$ powder, the $KTaO_3$ powder is mixed with KF powder and heated for 5 hours in an oxygen environment at $700^\circ C$. K_2TaO_3F is formed by solid-phase reaction by heating. K_2TaO_3F is dissolved in water and stayed for 12 hours after heating at $80^\circ C$. By agitating a solution, water is evaporated, and sediments are collected by filtration after verifying sedimentation in the solution. At this time, aqueous washing repeats and then HF components are removed. The produced powder is micronized $KTaO_3$ powder and an average particle diameter is about $10\mu m$.

[0041] The pressure of $5kg/mm^2$ is applied to the micronized $KTaO_3$ powder by an uniaxial pressing machine. Then, a pellet with a 30mm diameter and a thickness of 10mm is molded. After putting this pellet into a platinum container and covering with the micronized $KTaO_3$ powder, it is heated for 10 hours

in an oxygen environment at 1000°C in an electric furnace. After natural cooling, a transparent pellet which shrinks by sintering to a 20mm diameter and a 6mm thickness is taken out. The pellet is covered with the same constituent powder,
5 in order to inhibit K_2O to evaporate from the pellet surface. The vapor pressure of K_2O may be kept above the equilibrium vapor pressure by putting powder materials containing K_2O inside the electric furnace.

[0042] The obtained pellet is examined under a microscope
10 to find that it is a polycrystal with an average crystal grain of 50-100 μm . Optical transmittance and a refractive index of the pellet is almost the same as the above-referenced KT, and birefringence of the pellet is below the measuring limit. A SIL is produced as described in Example 1 by quarrying 1.2mm
15 cube from this pellet. When a recording density is estimated by the evaluation equipment for DVD recording, the recording density equals to that of the DVD recorder as described in Example 1. Since a crystal material is a cubic crystal, an optically isotropic lens may be produced by either of single
20 crystal materials or polycrystalline materials.

[0043] Although a powder production method with a solid-phase reaction is described in Example 8, a powder production method such as a sol-gel method, a coprecipitation method and the like may be used. Also, in order to suppress
25 oxygen deficiency of crystal materials as possible, an oxygen environment for sintering or an oxygen environment which partial pressure of oxygen is greater than equilibrium partial

pressure is desirable.

[0044] By adding Nb_2O_5 in the process of producing polycrystalline materials, polycrystalline materials of KTN may be produced. A polycrystalline KTN has optically isotropic characteristics and a sintered density reaches almost 100% without defects such as a void.